



Artificial Oxide Heterostructures with Tunable Band Gap

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TEMPLE UNIVERSITY-OF THE COMMONWEALTH SYSTEM OF HIGHER EDUCATION

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Final report of the air force research laboratory project “Artificial oxide heterostructures with tunable band gap”

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Abstract

We have performed the joint experimental and theoretical studies towards the realization of tunable band-gap in artificial oxide multiferroic thin-films and heterostructures under external stimulus such as epitaxial strain. In multiferroic oxides, the magnetic exchange interaction is crucially dependent on the hybridization between the magnetic cation and its surrounding oxygen cage. The above is an electronic band structure property including both the valence and conduction bands, as well as the band gap. In addition, the magnetic interaction can be also strongly coupled to the ferroelectric polar distortion through the metal-oxygen bonds angles and bonding lengths, which are sensible to the external stimulus. By using the coupling between magnetic and the ferroelectric orderings, the band structure, gap as well as the magnetic properties can be tuned by the epitaxial strain. We showed the tunable optical dielectric functions from the surface reconstruction and the epitaxial strain in CaMnO_3 films by epitaxial growth. We also demonstrated that the functional properties in hexagonal LuFeO_3 have its electronic and structural origins and can be tuned by epitaxial strain. Finally, we also proposed new design rule for room temperature artificial perovskite superlattice, which is closed related with the current topic.

Research details

Surface- and strain-tuning of the optical dielectric function in epitaxially grown CaMnO_3

From year 2013 to year 2016, the main efforts of our team have been focused on the tunable band gap and band structures in epitaxial grown CaMnO_3 . The efforts have been devoted to (1) the thin film growth; (2) the tunable optical dielectric function measurement; (3) the theoretical computation and understanding the mechanism in the experimental observations. The details have been summarized in the following.

Our thin-film experimental group under the leadership of Prof. Xiaoxing Xi at physics department of Temple University has grown the perovskite CaMnO_3 films under different epitaxial strain conditions by using different substrates in the growth. Epitaxial CaMnO_3 thin films were grown by pulsed laser deposition using a KrF excimer laser ($\lambda = 248$ nm) on single-crystal (001)-oriented SrTiO_3 (STO), LaAlO_3 (LAO), 2 and SrLaAlO_4 (SLAO) purchased from Crystec. The well controlled coherent growth condition and the good quality of the films are reported in Figure. 1. In order to characterize the optical properties of CaMnO_3 thin films, the Variable-angle spectroscopic ellipsometry was performed by Prof. John Spanier's group (one of our PIs at Drexel material department) at room temperature in ambient atmosphere with an electronically controlled rotating compensator and Glan-Taylor polarizers. The above measurements were performed at multiple angles between 65-75 degrees and in the spectral range of 247 to 1000 nm with a resolution of 1.6 nm. Measurement of the components of linearly polarized reactivity at each selected wavelength were used to obtain the ellipsometric parameters. To determine imaginary part of dielectric function for CaMnO_3 we assume a four-layer optical medium comprised of a homogeneous isotropic film layer on a semi-infinite bulk, incorporating surface roughness, in vacuum. The experimental dielectric function is reported in Figure 2. The dielectric functions of the CMO thin-films under different strain conditions in optical measurement were computed by the theoretical group of Xifan Wu at physics department of Temple University. The first-principles calculations were performed by using density functional theory as implemented in the Vienna Ab Initio Simulation Package. The electron exchange and correlation were approximated by the generalized

gradient approximation revised for solid and we adopted an effective on-site Coulomb repulsion $U - J = 3.0$ eV for the d orbitals of Mn atoms.

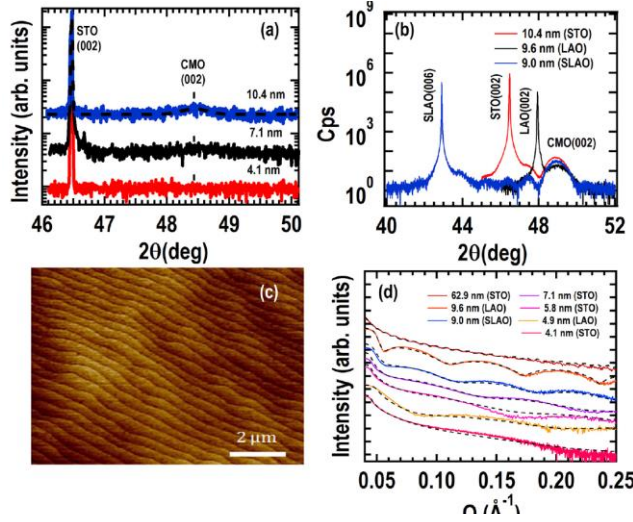


Figure 1. (a) GIXRD, (b) XRD, (c) AFM, and (d) XRR data of CMO films with varying thickness grown on different substrates. (a) GIXRD spectra of a 10.4, 7.1, and 4.1 nm thick CMO film grown on STO substrates. The film peak is at 48.44 degrees. Fitting is shown (dashed line) for the 10.4 nm film. (b) The 2θ - x XRD scan of CMO thin films (~ 10 nm in thickness) on each substrate. Substrate peaks and film peaks (48.93 degrees) are identified. (c) AFM of the 10.4 nm thick CMO film on STO showing the underlying terraces from the annealed STO substrate, indicating an atomically flat film. (d) The XRR spectra for films with different thicknesses on STO, LAO, and SLAO substrates. Fitting is given for each trace (dashed line).

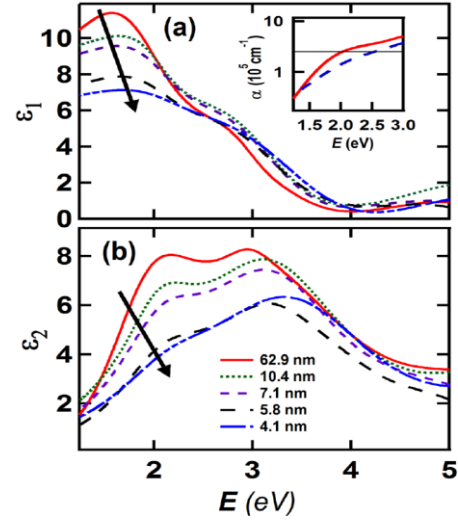


Figure 2. (a) Real, and (b) imaginary part, of the experimentally determined complex frequency-dependent dielectric function as a function of photon energy for different CMO film thicknesses on STO (001). The arrow denotes films of decreasing thickness, as specified in Table I. The thinnest two films are strain coherent, as the films increase in thickness they begin to strain relax. The inset shows a for the 62.9 nm (solid) and 4.1 nm (dotted) films plotted on a semi-log scale over the range 1.2–3 eV. At a fixed value of absorption (solid line shows optical absorption = $2.5 \times 10^5 \text{ cm}^{-1}$), there is as much as a ~ 0.5 eV spectral shift in the photon energy for decreasing film thickness.

Based on the above joint experimental and theoretical analysis, in a recently published paper on *Applied Physics Letters*¹ in 2015, we reported a strong thickness dependence of the complex frequency-dependent optical dielectric function over a spectral range from 1.24 to 5 eV in epitaxial CaMnO_3 (001) thin films on SrTiO_3 (001), LaAlO_3 (001), and SrLaAlO_4 (001). A doubling of the peak value of the imaginary part of dielectric function and spectral shifts of 0.5 eV for a given magnitude of absorption are observed. On the basis of experimental analyses and first-principles density functional theory calculations, contributions from both surface states and epitaxial strain to the optical dielectric function of CaMnO_3 are seen. Its evolution with thickness from 4 to 63 nm has several regimes. In the thinnest, strain-coherent films, the response is characterized by a significant contribution from the free surface that dominates strain effects. However, at intermediate and larger thicknesses approaching the bulk-like film, strain coherence and partial strain relaxation coexist and influence the dielectric function.

Our results pointed out that in the thinnest films the surface contribution is crucial in giving rise to the observed tunable dielectric function properties in optical measurements, whereas in the thicker films progressive partial and full strain relaxation dominates the evolution of dielectric function. The interplay among thickness-driven large surface and substrate-induced strain contributions to dielectric function in an epitaxial perovskite oxide thin film in its non-magnetic phase holds promise for a novel route to thickness-induced engineering of optical properties.

Structural and electronic origin of the magnetic structures in hexagonal LuFeO₃

Besides our main efforts as a joint experimental and theoretical team in studying the tunable band structures and band gap in the CaMnO₃ films by dielectric function measurement, we also study tunable functional properties such as ferroelectric polarization and ferromagnetic moment in the improper oxide multiferroic materials. These tunable properties are closely associated with the band structure properties. We then focus also on the recently discovered multiferroic of hexagonal LuFeO₃. It provides a rare case multiferroic material in which spontaneous electric and magnetic polarizations coexist. On one hand, ferroelectricity appears below $T_C=1050$ K resulting from a $P6_3/mmc \rightarrow P6_3cm$ structure distortion, which can be decomposed in terms of three phonon modes. On the other hand, spin frustration in it presents rich magnetic phases. Intriguingly, below the Neel temperature, magnetic order in hexagonal LuFeO₃ transits from B_2 to A_2 as shown in Figure 3 (b) at 130 K, resulting in a weak ferromagnetism due to the Dzyaloshinskii-Moriya and single-ion anisotropy mechanism.

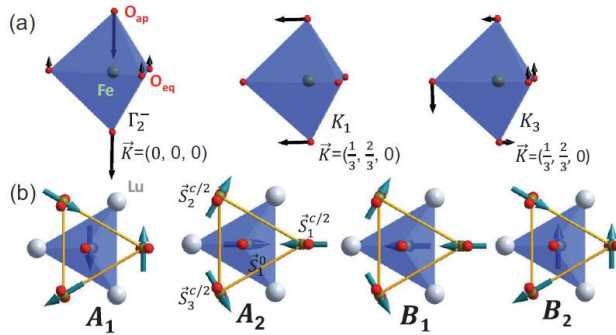


Figure 3. (a) Displacement patterns of the FeO₆ local environment (trigonal bipyramid) in the three phonon modes that freeze in the $P6_3/mmc \rightarrow P6_3cm$ structural transition in hexagonal LuFeO₃. The arrows indicate the relative displacement of the atoms. K is the wave vector of the modes in the reciprocal space of the $P6_3/mmc$ structure. (b) Four independent spin structures (A_1 , A_2 , B_1 , and B_2) of the 120-degree magnetic orders viewed along the c axis. The arrows indicate the spins on the Fe sites.

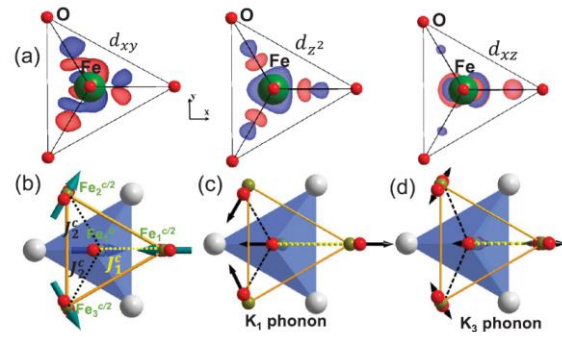


Figure 4. (a) Representative d_{xy} , d_{z^2} , and d_{xz} -like Wannier functions viewed from $[001]$ direction. (b) Illustrations of two independent super-super-exchange paths J_{c1} and J_{c2} between Fe0 at $z/c = 0$ and three neighboring iron ions Fe₁, Fe₂, and Fe₃ at $z/c = 1/2$. (c) Atomic displacements of the K_1 phonon mode. (d) Atomic displacements of the K_3 phonon mode, viewed from $[001]$ direction.

In order to understand the electronic origin of the relatively high Neel temperature in hexagonal LuFeO₃ and the structural origins of the stabilized A_2 phase that makes the ferromagnetic ground state at low temperature. First, the theoretical group of Xifan Wu at Temple University has applied an extended Kugel-Khomskii model for superexchange interactions based on localized Wannier functions. While the antiferromagnetic exchange coupling is dominated by the intralayer superexchange, the model clearly showed that the singly occupied d_{z^2} orbital in hexagonal LuFeO₃ greatly increases the exchange coupling compared with the empty d_{z^2} in LuMnO₃. The interlayer exchange, although much weaker in magnitude, is key to the stabilization of ferromagnetic ground state. Our first-principles calculations showed that stabilization of A_2 phase is strongly coupled to the K_1 phonon mode and only weakly dependent on K_3 mode. Our theory indicates that the atomic displacements of K_1 phonon mode is responsible for the stabilization of A_2 phase with ferromagnetic moment.

Our theoretically predicted scenario is then confirmed by our experimental collaborators at University of Nebraska-Lincoln with the x-ray diffraction and x-ray absorption measurements. XAS measurements suggest that the K_1 mode undergoes a gradual change at low temperature. Our joint theoretical and experimental discoveries have been published in *Physics Review B*². Our studies pointed out the important structural origin of the ferromagnetic ground state of A_2 phase that is responsible for the appearance of spontaneous magnetization, is identified by theory and verified by x-ray diffraction and absorption experiments. It also provides important foundations for the future studies of magnetoelectric coupling and the tunable band gaps properties in this material.

Effects of biaxial strain on the improper multiferroicity in hexagonal LuFeO_3 films

Elastic strain is expected to play an important role in tuning the property of improperly multiferroic hexagonal ferrites. In the year of 2016, our theoretical group carried out first-principles calculations to elucidate the structural distortions at the atomic level under the epitaxial strains ranging from -2% to 2% in hexagonal LuFeO_3 films. Based on the relaxed structures, the mode decompositions performed based on the group theory. Our theoretical calculations show that the mode amplitude Q_{K3} is enhanced (reduced) by the applied compressive (tensile) epitaxial strains. As schematically shown in Figure 5(c), it can be seen that oxygen atom at the center of the trimer that shared by three bi-pyramids is moving up, while the other oxygen atoms in the bases of the three bi-pyramids are all moving downwards under compressive strain. As a result, the mode amplitude Q_{K3} is increased. However, we found that the tunability of polarization by epitaxial strain is much less than the conventional ferroelectric such BaTiO_3 . The weak ferromagnetism originates from both the DM interaction and single ion anisotropy. The DM vector $\mathbf{D} \sim |\mathbf{r}_{\text{Fe-Fe}} \times \boldsymbol{\delta}_z|$ in which $\mathbf{r}_{\text{Fe-Fe}}$ is the displacement vector between the two iron atoms and $\boldsymbol{\delta}_z$ is the displacement vector along [001] direction for the oxygen atom shared by three bi-pyramids in the trimer shown in Figure 5(a) respectively. Since $\boldsymbol{\delta}_z$ is closely associated with the trimerization measured by the Q_{K3} , the weak ferromagnetism was found to intrinsically related to the K_3 mode. However, the compressive strain also brings the two Fe atom closer which reduces the displacement vector $\mathbf{r}_{\text{Fe-Fe}}$ more rapidly as shown in Figure 5(a), which actually reduces magnitude of cross product of the DM vector. As a result, the canting ferromagnetic moment is rather decreased as shown in Figure 6(b).

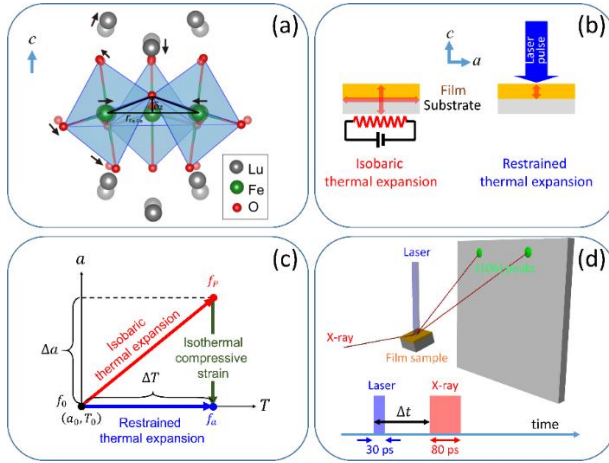


Figure 5. (a) Model of the K_3 lattice distortion pattern and the atomic displacement under a compressive strain. (b) Schematics of the strain generated by the restrained thermal expansion in comparison with the isobaric thermal expansion. (c) Illustration of the restrained thermal expansion and isobaric thermal expansion in the (a, T) space, where a is the in-plane lattice constants, T is temperature, f is a general physical property. (d) Illustration of the experimental setup for the pump (laser) and probe (x-ray) measurements.

Our theoretical calculation is consistent with the experimental work performed at physics department of University of Nebraska-Lincoln. Prof. Xu's group employed a method of studying the effect of biaxial strain in thin films involving restrained thermal expansions [Figure 5(b)]. In general, thermal strain may be generated in a material in all crystalline dimensions in isobaric thermal expansion. In an epitaxial thin film, on the other hand, if the film is selectively heated, the in-plane dimensions of the film is restrained by the substrate, while the out-of-plane dimension is free to expand (restrained thermal expansion). By comparing the material properties in the isobaric and restrained thermal expansions, the effect of isothermal compressive strain at a higher temperature can be obtained [Figure 5(c)]. Using the method of restrained

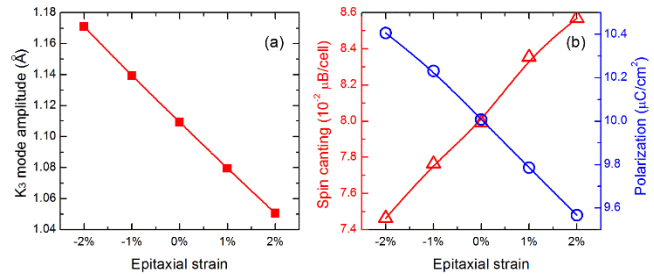


Figure 6. Effect of the biaxial strain $\frac{\Delta a}{a}$ on the K_3 distortion (a) and on the electric polarization and the weak ferromagnetic moment, calculated using the density functional theory.

thermal expansion, they have studied the strain effect on the K_3 ferrodistortion in LuFeO_3 . Experimentally, they found that the biaxial strain in the basal plane of LuFeO_3 does significantly couple to the ferrodistortive structural distortion, which agrees exactly with the first principle calculation.

We have submitted our results to *Physics Review Letter*³ which is currently under active review. These findings are important for understanding on the strain effect as well as the coupling between the lattice and the improper multiferroicity in LuFeO_3 . The experimental elucidation of the strain effect in LuFeO_3 films also demonstrates that the restrained thermal expansion combined with first-principles theories can be a viable method to unravel the strain effect in many other epitaxial thin film materials.

Theoretical design of room temperature multiferroism in CaTcO_3 by interface engineering

Besides the joint experimental and theoretical work as described in the above, the Wu's theoretical group at Temple University has been also taking an effort in the theoretical design of functional multiferroic materials, which could be used as the material for tunable band gap with applied stimulus. In the year of 2014 and 2015, we used the density functional theory to perform a systematic investigation of the structural instabilities and distortions in ATcO_3 ($A = \text{Ca, Sr, Ba}$), in which a variety of structural instabilities has been identified. At ground state of CaTcO_3 and SrTcO_3 , the antiferrodistortive modes associated with oxygen octahedral rotations are found to originate from the zone boundary phonon instabilities at Brillouin zone boundary points. An antipolar structure is found in CaTcO_3 and SrTcO_3 originating from the antiferroelectric instability at X point and a trilinear coupling with M and R instabilities that lowers the free energy. A ferroelectric instability is discovered in only CaTcO_3 , which is however suppressed by the large anti-ferrodistortive mode at the ground state. In the latter case, we theoretically investigate the interface induced ferroelectric polarization in $\text{CaTcO}_3/\text{BaTcO}_3$ superlattice. By taking advantage of the intrinsic structural instabilities in CaTcO_3 , including antipolar and polar ones, we propose that the ferroelectricity can be induced at the interfaces of the superlattice by two interface engineering approaches. First, the electric polarization can be induced by the mismatch of antipolar structure between BaTcO_3 and CaTcO_3 . Second, the large oxygen octahedral rotation will be suppressed at the interface which can be used to enhance the intrinsic ferroelectric mode. This picture is confirmed by our first-principles' calculations and the analysis of $\text{CaTcO}_3/\text{BaTcO}_3$ superlattices. Finally, the Neel temperature of the superlattice is calculated to be ~ 816 K by a following Monte Carlo simulation. Our theoretical predictions clearly suggest that the room temperature multiferroism can be achieved in CaTcO_3 by interface engineering.

The results suggested new routes to engineer non-polar perovskite into new room temperature multiferroic materials. The theory has been published in the journal of *Computational Materials Science*⁴. The work is now awaiting for experimental confirmation.

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Abstract

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